

Spin reorientation in Na-doped BaFe_2As_2 studied by neutron diffraction

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We have studied the magnetic ordering in Na doped BaFe_2As_2 by unpolarized and polarized neutron diffraction using single crystals. Unlike previously studied FeAs-based compounds that magnetically order, $\text{Ba}_{1-x}\text{Na}_x\text{Fe}_2\text{As}_2$ exhibits two successive magnetic transitions: For $x=0.35$ upon cooling magnetic order occurs at ~ 70 K with in-plane magnetic moments being arranged as in pure or Ni, Co and K-doped BaFe_2As_2 samples. At a temperature of ~ 46 K a second phase transition occurs, which the single-crystal neutron diffraction experiments can unambiguously identify as a spin reorientation. At low temperatures, the ordered magnetic moments in $\text{Ba}_{0.65}\text{Na}_{0.35}\text{Fe}_2\text{As}_2$ point along the c direction. Magnetic correlations in these materials cannot be considered as Ising like, and spin-orbit coupling must be included in a quantitative theory.

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There are two promising explanations for the appearance of high-temperature superconductivity in FeAs-based materials [1]. Orbital fluctuations may result in a s^{++} superconducting state [2, 3] and can reflect the fact that highest superconducting transition temperatures arise in materials with almost ideal FeAs_4 tetrahedrons [4] and, thus, with highest orbital degeneracy. On the other hand, there are strong magnetic fluctuations associated with the antiferromagnetic (AFM) order in the parent compounds which can explain a s^\pm superconducting state [5].

Magnetism and orbital degrees of freedom are closely tied in FeAs-based compounds. Although the structural distortion accompanying AFM order in the parent materials remains small [6–8] its electronic signatures are rather strong as seen in the anisotropic resistance [9, 10], in angle-resolved photoemission studies (ARPES) [11, 12] or optical spectroscopy [13]. In addition the magnon dispersion in the AFM state is fully anisotropic [14] inspiring theoretical models of orbital order driving magnetic interaction similar to those applied to manganates [15–17]. Studying the interplay between orbital and magnetic degrees of freedom seems crucial for the understanding of FeAs-based materials.

Here we focus on spin-space anisotropies arising from the spin-orbit coupling between spin and orbital moments and which thus allow for a direct view on orbital contributions. In AFM BaFe_2As_2 , polarized neutron scattering shows that it costs more energy to rotate the magnetic moment within the planes than perpendicular to them [18]. Magnetic anisotropy clearly persists into the superconducting range of the phase diagrams [19–23].

So far all AFM ordered FeAs-based compounds exhibit a single magnetic transition to a magnetic structure where moments are aligned parallel to the in-plane

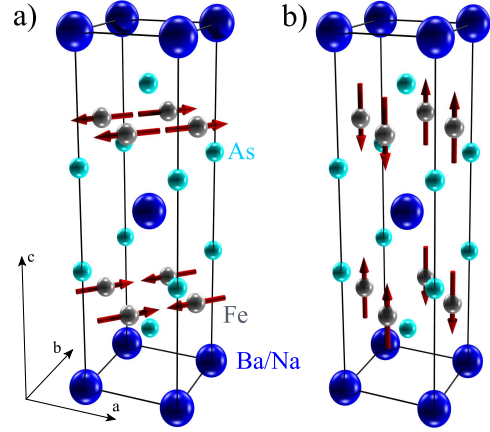


FIG. 1: (Color online) Magnetic structure of $\text{Ba}_{1-x}\text{Na}_x\text{Fe}_2\text{As}_2$ in the AFM1 phase $46 < T < 70$ K and in the AFM2 phase at low temperature, $T < 46$ K. Note that the fourfold axis is broken in both cases by the in-plane component of the magnetic propagation vector chosen as $(0.5, 0.5, 1)$.

component of the magnetic propagation vector, which is $(0.5, 0.5, 1)$ [6–8] (we use reduced lattice units with respect to $\frac{2\pi}{a}$ and $\frac{2\pi}{c}$ with $a \sim 3.9$ and $c \sim 13.1$ Å). Very recently a second magnetic transition was reported for Na-doped BaFe_2As_2 and interpreted as a microscopic superposition of the two equivalent ordering schemes, with propagation vectors $(0.5, 0.5, 1)$ and $(-0.5, 0.5, 1)$, into a two- k structure [24]. We have studied single crystalline samples of $\text{Ba}_{1-x}\text{Na}_x\text{Fe}_2\text{As}_2$ at various doping levels finding clear evidence for a reorientation of the spins occurring at intermediate doping at low temperature. This spin reorientation towards alignment of the magnetic moments parallel to the c direction agrees with the anisotropies

observed in pure and in Co-doped BaFe_2As_2 .

Single crystals of $\text{Ba}_{1-x}\text{Na}_x\text{Fe}_2\text{As}_2$ were grown using a self-flux high temperature solution growth technique. Details of sample growth and characterization can be found in reference [25]. Due to the air-sensitivity of $\text{Ba}_{1-x}\text{Na}_x\text{Fe}_2\text{As}_2$ all synthesis procedures as well as the mounting and orienting of the crystals into the sample cans were done in an Ar box. Lattice parameters of the large crystals were determined by neutron diffraction using the 3T1 diffractometer at the Orphée reactor in Saclay ($k_i=2.66 \text{ \AA}^{-1}$). There are two reports on the phase diagram of $\text{Ba}_{1-x}\text{Na}_x\text{Fe}_2\text{As}_2$ in the literature [26, 27], both based on polycrystalline samples. The lattice constants and magnetic properties determined with our single crystals fit to the results reported by Cortes-Gil et al. [27] while there seems to be an offset in the Na-content when comparing with the data described by Avci et al. [26]. Neutron scattering experiments aiming at the characterization of the magnetic structure as function of temperature were performed at the PANDA triple-axis spectrometer (Maier-Leibnitz Zentrum, Garching, $k_i=1.55$ and 2.57 \AA^{-1}), at 3T1 and at the 4F1 triple-axis spectrometer (both at the Orphée reactor)). On the 4F1 spectrometer we used a neutron beam with $k_i=2.57 \text{ \AA}^{-1}$ polarized by a bender and analyzed the final polarization by a Heusler crystal. We performed longitudinal polarization analysis by guiding the neutron spins with a set of Helmholtz coils. In all experiments either pyrolytic graphite or Be filters were used to suppress higher order contaminations and crystals were oriented in the $[110]/[001]$ scattering planes.

Fig. 1 shows the crystal and the magnetic structures of $\text{Ba}_{1-x}\text{Na}_x\text{Fe}_2\text{As}_2$. Fig. 1(a) corresponds to the alignment of magnetic moments parallel to the layers which is found in most FeAs-based compounds [6–8]. One has to distinguish three orthogonal directions: one is defined by the in-plane component of the magnetic propagation vector (chosen here as $(0.5,0.5,1)$), $[1,1,0]$ labelled longitudinal in-plane, the second is $[-1,1,0]$ transversal in-plane, and the third $[0,0,1]$ out-of-plane. Fig. 2 shows the temperature dependence of nuclear and magnetic Bragg reflections measured on $\text{Ba}_{0.65}\text{Na}_{0.35}\text{Fe}_2\text{As}_2$. The magnetic peaks $(0.5,0.5,1)$ and $(0.5,0.5,3)$ start to increase in intensity when cooling below the first magnetic transition at 70 K. There is a second magnetic transition at lower temperature where both peaks further increase, but this low-temperature intensity enhancement is much stronger for the $(0.5,0.5,1)$ Bragg peak. Already this different behavior of intensities at the two transitions indicates a change in the magnetic structure at the lower transition. Fig. 2 also shows the temperature dependence of two nuclear Bragg peaks which both show anomalies at the two magnetic transitions. This impact of the magnetic ordering on the nuclear structure underlines close coupling but clear interpretation of these structural effects is not obvious. The emergence of orthorhombic domains

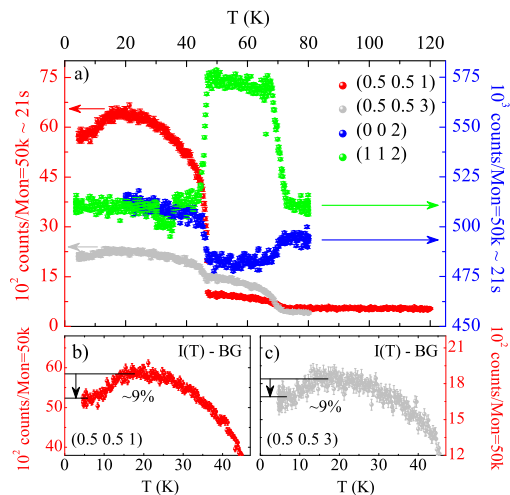


FIG. 2: (Color online) (a) Temperature dependence of magnetic and nuclear Bragg reflection intensities in $\text{Ba}_{0.65}\text{Na}_{0.35}\text{Fe}_2\text{As}_2$ measured on the PANDA spectrometer. Note that the magnetic, $(0.5,0.5,1)$ and $(0.5,0.5,3)$, and nuclear, (112) and (002) , Bragg peaks refer to different scales respectively. (b) and (c) show an enlargement of the low-temperature range illustrating the reduction of magnetic intensities in the superconducting state.

in the magnetically ordered phase has strong impact on extinction and multiple diffraction conditions of nuclear Bragg reflections [19]. The up and down shifts of nuclear intensities at the two transitions indicates that the structural changes appearing at the high-temperature magnetic transition get suppressed at the lower transition. The anomalies in the nuclear Bragg peaks are not perfectly sharp as it can be expected for an extinction effect at a structural transition, but the width of the anomaly amounts only to a few degrees K so that any local variation of the Na doping must be very limited.

There is a third anomaly visible in the temperature dependencies of the magnetic Bragg peaks. At low temperature in the superconducting state both magnetic Bragg peaks decrease in intensity, see Fig. 2. The relative reduction in the two magnetic peaks is identical indicating that this sample exhibits a single magnetic phase at low temperature whose ordered moment gets reduced in the superconducting state.

In order to quantitatively analyze the change in magnetic structure we have measured integrated intensities on PANDA by performing rocking scans. The integrated intensities were corrected for the Lorentz factor and are traced in Fig. 3 against the q_l component. Neutron diffraction only measures the magnetic moment perpendicular to the scattering vector. Any magnetic diffraction intensity is weighted with a factor $\sin^2(\alpha)$ with α being the angle between the magnetic moment and the scattering vector. By enhancing the q_l component in the mag-

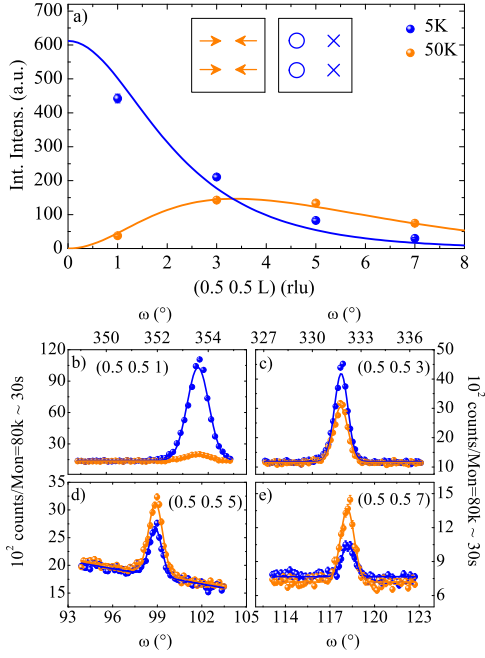


FIG. 3: (Color online) Part (a) shows the l dependence of magnetic integrated intensities in the AFM1 and AFM2 phases. The lines are model calculations for the magnetic structures shown in Fig. 1 with moments parallel to the a and c directions. (b-e) Rocking scans across the magnetic Bragg peaks $(0.5, 0.5, l)$ with $l = 1, 3, 5, 7$, measured on the PANDA spectrometer at 5 and 50 K, in the AFM2 and AFM1 phases, respectively.

netic Bragg reflection $\mathbf{Q} = (0.5, 0.5, q_l)$ a magnetic moment aligned along the c direction contributes less and a moment along the in-plane component of the Bragg scattering vector, i.e. along $[110]$, contributes more strongly. In addition the magnetic form factor depends on the length of the scattering vector in an isotropic approximation and reduces the magnetic scattering at larger Q values. Both effects can be calculated for $\mathbf{Q} = (0.5, 0.5, q_l)$ and are included in Fig. 3 for the usual magnetic arrangement with moments aligned along the in-plane component of the propagation vector (orange line, index a) and for out-of-plane alignment (blue line, index c):

$$I_{a,c}(0.5, 0.5, q_l) \propto [f(|Q|) \cdot m \cdot \sin(\alpha_{a,c})]^2 \quad (1)$$

$$\sin(\alpha_a) = \frac{q_l \cdot \frac{2\pi}{c}}{Q}; \quad \sin(\alpha_c) = \frac{\frac{1}{\sqrt{2}} \cdot \frac{2\pi}{a}}{Q} \quad (2)$$

At 50 K, i.e. in the magnetic phase at higher temperature, the integrated intensities are perfectly described by the in-plane arrangement, while the low temperature data agree well with the out-of-plane alignment. Some minor deviations may arise from an anisotropic magnetic form factor or from a small fraction of the sample staying in the AFM1 phase (fitting the data in Fig. 3 (a))

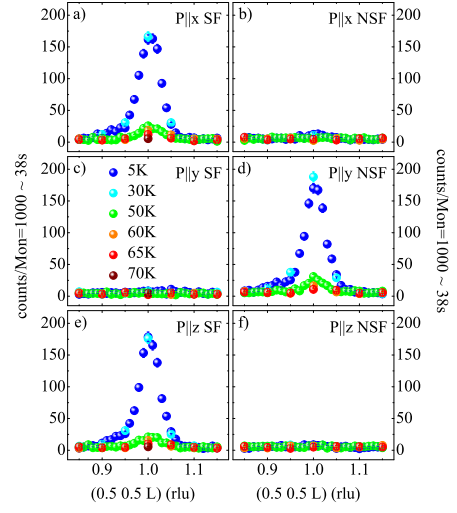


FIG. 4: Results of polarized neutron-diffraction experiments on the 4F spectrometer. Scans were performed across the $(0.5, 0.5, 1)$ magnetic Bragg peak in both the SF and NSF channel for the neutron polarization along the x , y and z directions. There is no indication for any scattering associated with magnetic moment pointing in the $[1, -1, 0]$ direction.

with both phases and equal moments indicates only 13% AFM1).

We may conclude that $\text{Ba}_{0.65}\text{Na}_{0.35}\text{Fe}_2\text{As}_2$ exhibits a spin reorientation transition at ~ 46 K with moments aligned along the c direction at low temperature. The different intensity changes observed at different $(0.5, 0.5, q_l)$ peaks are furthermore illustrated in the lower parts of Fig. 3. The gain in intensity is most pronounced at $(0.5, 0.5, 1)$ where the geometry factor strongly hampers the observation of the magnetic peaks in the AFM1 phase, $\sin^2(\alpha_a) = 0.16$, while out-of-plane moments contribute with an almost full geometry factor in AFM2, $\sin^2(\alpha_c) = 0.84$. The scans across $(0.5, 0.5, 5)$ and $(0.5, 0.5, 7)$ show that these intensities even decrease at the lower transition because the geometry factor is strongly reduced at the spin reorientation.

We have strengthened our conclusion about the spin reorientation in $\text{Ba}_{0.65}\text{Na}_{0.35}\text{Fe}_2\text{As}_2$ by experiments using polarized neutron scattering, see Fig. 4. The sample was again mounted with the $[110]/[001]$ scattering plane. The polarization analysis adds the additional selection rule that magnetic scattering contributes to the spin-flip (SF) signal if the magnetic contribution is perpendicular to the axis of the polarization analysis, while the component parallel to the polarization axis contributes to the non-spin-flip (NSF) scattering. One may analyze the SF and NSF signals for setting the neutron-polarization axis parallel to the scattering vector (labelled x -direction), parallel to the $[110]$ direction (labelled z -direction, as it is perpendicular to the scattering plane) and perpendicular to

these two directions (labelled y -direction). Fig. 4 shows the measurement obtained with the $\text{Ba}_{0.65}\text{Na}_{0.35}\text{Fe}_2\text{As}_2$ -crystal at the (0.5,0.5,1) reflection, measurements at (0.5,0.5,3) yielded the same results. For x -axis polarization analysis the scattering appears almost entirely in the SF channel, as it is expected for a magnetic signal. The small leakage in the NSF channel is due to imperfection of the polarization analysis, but note that the flipping ratio of the total experimental setup is quite high, ~ 31 . Concerning the other two polarization directions the signal is entirely found in the NSF channel in y -direction and in the SF channel in the z -direction. These findings document that there is no ordered magnetic moment along the $[110]$ direction (in-plane transversal) neither in the high-temperature nor in the low-temperature phase. The magnetic structure with the same propagation vector (0.5,0.5,1) but with transversal in-plane moments is not observed in $\text{Ba}_{0.65}\text{Na}_{0.35}\text{Fe}_2\text{As}_2$. The magnetic moment thus always stays within the scattering plane. Taking into account the q_l dependence of the signal, the spin reorientation from $[110]$ to $[001]$ directions is the only possible explanation of our single-crystal data.

We have also studied another crystal with a 35% Na doping as well as ones with 25 and 39%. All these crystals show qualitatively the same two magnetic transitions and the spin reorientation. However, the transition temperatures depend on the doping and at 25% Na and 39% Na contents the low temperature phase transition seems to stay incomplete indicating the limits of the stability of the c aligned magnetic phase. A crystal with a doping content of 40% did not show any static magnetic ordering indicating a rapid suppression of the magnetic phase.

Avci et al. reported on powder neutron diffraction experiments on a sample with nominal composition $\text{Ba}_{0.76}\text{Na}_{0.24}\text{Fe}_2\text{As}_2$ [24] which also indicate a second magnetic transition in good agreement with our results. This powder sample shows reentrance into an almost tetragonal phase, but the powder data and the fact that 40% of the sample did not transform prohibited an unambiguous determination of the magnetic structure. The results in [24] were discussed as evidence for a $2k$ magnetic structure associated with the simultaneous condensation of both nematic order parameters. Since our results clearly document the spin reorientation as the main element of the low-temperature magnetic transition the possible existence of a $2k$ structure needs further analysis, because such structure implies non-magnetic or weakly magnetic sites for c -aligned moments, and because the spin reorientation can at least partially explain the suppression of the orthorhombic splitting.

The spin reorientation observed in $\text{Ba}_{1-x}\text{Na}_x\text{Fe}_2\text{As}_2$ is in agreement with the anisotropies found in pure and in doped BaFe_2As_2 [18, 19, 23]. The analysis of the gaps of magnetic excitations in pure BaFe_2As_2 indicates that rotating the moment into the c direction corresponds to the lower anisotropy energy in contrast to the expectation

for a layered magnet. Na substitution seems to further reduce the c -axis anisotropy energy so that it becomes the ground state ordering for 35% of doping. The change in the direction of the magnetic moment must be associated with a slightly different orbital arrangement.

The ARPES experiments in the AFM phase of BaFe_2As_2 indicate significant lowering of the electronic band associated with d_{xz} orbitals (in the coordinates with a along the AFM ordered moment) [11]. In a simple localized picture, orbital ordering with enhanced occupation of the d_{xz} orbital can mix through spin-orbit coupling with other orbitals yielding a finite orbital moment. Linear combinations with the other t_{2g} orbitals $d_{xz} + i \cdot d_{yz}$ and $d_{xz} + i \cdot d_{xy}$ yield orbital moments along z and x , respectively, which are the two directions of ordered moments in AFM FeAs-materials. The enhanced occupation of the d_{xz} orbital observed in ARPES qualitatively agrees with the observed ground states in the AFM phases but a quantitative analysis of the spin-orbit coupling in the metallic AFM state is highly desirable. Avci et al. [26] find that the FeAs layers are more extended in c direction in the Na-doped series, which favors the orientation of magnetic moments in c direction giving further support to our orbital physics argumentation.

The different magnetic ground state seems not to be reflected by a quantitative change in the superconducting T_c as compared to the K-doped series [25–28]. This fact is a challenge for any quantitative pairing theory in particular one based on orbital fluctuations. The spin reorientation, furthermore, is relevant for discussing the possible role of the nematic phase [29]. At least the breaking of the fourfold axis due to the in-plane alignment of the ordered moment seems not to possess a significant effect on the superconducting pairing. The two different magnetic arrangements appearing in FeAs-compounds, furthermore, indicate that the magnetic character in these materials is not Ising like. Instead orbital effects seems just to imply a magnetic hard axis (transversal in-plane).

The c -polarized phase seems, furthermore, to agree with the recent observation of a sharp low-energy resonance mode in optimally Co-doped BaFe_2As_2 , which arises in the c -polarized channel [19]. This experiment showed that this low-energy mode does not appear in the in-plane transversal direction, while a similar experiment on a Ni-doped BaFe_2As_2 sample [23] indicates that the low-energy mode also has some longitudinal in-plane character. Therefore, one may conclude that also in the doped superconducting compounds the lowest magnetic excitations are found in in-plane longitudinal and in c -oriented channels (easy plane) while in-plane transversal excitations lie higher in energy. The low-energy excitations in superconducting samples correspond thus to the two directions, where ordered moments are observed in the static phases.

So far only $\text{Ba}_{1-x}\text{Na}_x\text{Fe}_2\text{As}_2$ exhibits the c -polarized order, but the high-pressure phase recently reported in

K-doped BaFe_2As_2 could have the same character [30]. Magnetic order in the transversal in-plane direction is never observed and the corresponding magnetic response in the superconducting phase lies higher in energy underlining a common orbital effect. The transversal in-plane direction remains the magnetic hard axis independently of the presence of static magnetic order.

In conclusion we have shown that $\text{Ba}_{1-x}\text{Na}_x\text{Fe}_2\text{As}_2$ exhibits a spin reorientation at low temperature. The magnetic order in $\text{Ba}_{0.65}\text{Na}_{0.35}\text{Fe}_2\text{As}_2$ corresponds to that observed in most other FeAs-based magnetic materials for temperatures between 46 and 70 K, but at 46 K magnetic moments reorient along the c direction. This different magnetic structure seems not to be reflected in a modified superconducting pairing as superconducting transition temperatures in Na and in K doped systems are comparable. AFM and superconducting FeAs-based compounds exhibit evidence for the same and sizeable easy-plane anisotropy underlining the importance to take spin-orbit coupling into account.

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